Report for AOARD 11-4099

"Ion Source Development for a Compact Proton Beam Writing System II"

17 February 2012

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Abstract:

During this contract period the PI, a PhD student, a research fellow, a research technician and the collaborators have been working on this project. During the field trips to Delft University the team has gained valuable knowledge and have achieved the first results with the electron-impact ion source system. Through this exchange the team has now a realistic design for the ion source test bench which is being installed in Singapore. The first test beams with the conventional RF ion source have been achieved.

Electron beam calculations have been performed to evaluate different electron guns for their effectiveness in ionizing hydrogen gas molecules in our proposed configuration. At the same time the new proton beam writing beam line developed under grants AOARD 07-4017 & 09-4020, has been further improved and proton beams can now be focused down to 13 x 29 nm^2, some of the improvements are related to earlier projects: AOARD 06-4004 & AOARD 05-4037.

Introduction: ***Current status***

To overcome the diffraction constraints of traditional optical lithography, the next generation lithographies (NGLs) will utilize any one or more of EUV (extreme ultraviolet), X-ray, electron or ion beam technologies for producing sub-100nm

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14. ABSTRACT
The team has a realistic design for the ion source test bench which is being installed in Singapore. The first test beams with the conventional RF ion source have been achieved. Electron beam calculations have been performed to evaluate different electron guns for their effectiveness in ionizing hydrogen gas molecules in their proposed configuration. At the same time the new proton beam writing beam line developed under grants AOARD 07-4017 & 09-4020, has been further improved and proton beams can now be focused down to 13 x 29 nm2, some of the improvements are related to earlier projects: AOARD 06-4004 & AOARD 05-4037.

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features.

Electron beam lithography (EBL), a candidate for direct-write technology at nanodimensions has extensively been investigated for the last four decades However, high resolution lines and spaces in single step exposures for EBL are limited to about 20-30 nm levels due to proximity effects from high energetic secondary electrons initiating from adjacent and nearby features giving rise to structure broadening.

Perhaps the most under-developed and under-rated is the utilisation of ions for lithographic purposes. The three ion beam techniques, Proton Beam Writing (PBW), Focused Ion Beam (FIB) and Ion Projection Lithography (IPL) have the flexibility and potential to become leading contenders as NGLs [1]. Since the introduction of PBW in the Japanese governments road map for the nanotechnology business creation initiative (see appendix I) a large multinational corporation, Kobelco, has been pushing the development of compact accelerators for PBW applications. The Shibaura Institute of Technology (Tokyo, Japan) has recently received a 2M US$ grant from the Japanese government to develop PBW. In our Centre for Ion Beam Applications (CIBA), Physics Department, National University Singapore ***we established sub 100 nm proton beam focusing for protons down to 35x75nm^2*** [2] ***and have produced 3D high aspect ratio walls down to 22 nm in HSQ*** [3].The minimum obtainable feature size is expected to be in the nano meter range, due to the absence of proximity effects. In order to achieve nm sized features the proton probe has to be focused down to nm dimensions.

In CIBA we have been working on next generation systems for proton beam focusing. The success of a next generation PBW system depends on two main components: a <u>**stable high brightness source of MeV protons**</u> and a <u>high quality focusing lens system</u>. With the help of the US air force we have designed a new system for proton beam writing (AOARD 07-4017). The key characteristics are an improvement of the system demagnification. In initial experiments a beam has been focused down to 13 nm in x direction, closely matching the beam optical calculations.

Proton beam writing has the advantage of proximity free fabrication of high aspect-ratio nanostructures in photo-resist. Recently a proton beam size of 13 × 30 nm^2, has been achieved at a current of about 4 fA. The reduced brightness was measured to be about 10 A/m^{2}SrV [4]. For proton beam writing, the exposure dosage for photo-resist is typically 10-100 nC/mm^2. The beam resolution and writing time are limited by the low brightness RF ion source.

Experiment, Results and Discussion:

Ion source test bench setup

In order to optimize the current RF ion source used in the CIBA Singletron accelerator, we have been working on building an ion source test bench, with which we expect to improve the ion source brightness by about 10 times. This basic test setup will comprise of a gas handling system, high voltage and RF power supply to ionize the hydrogen gas molecules. The system will eventually be able to measure the proton beam current, energy spread and brightness. Initially the system will be tested at low voltage (several kV). The test-bench schematics are shown in figure 1. Next the test bench (Fig. 2) will be upgraded to include beam brightness measurements.

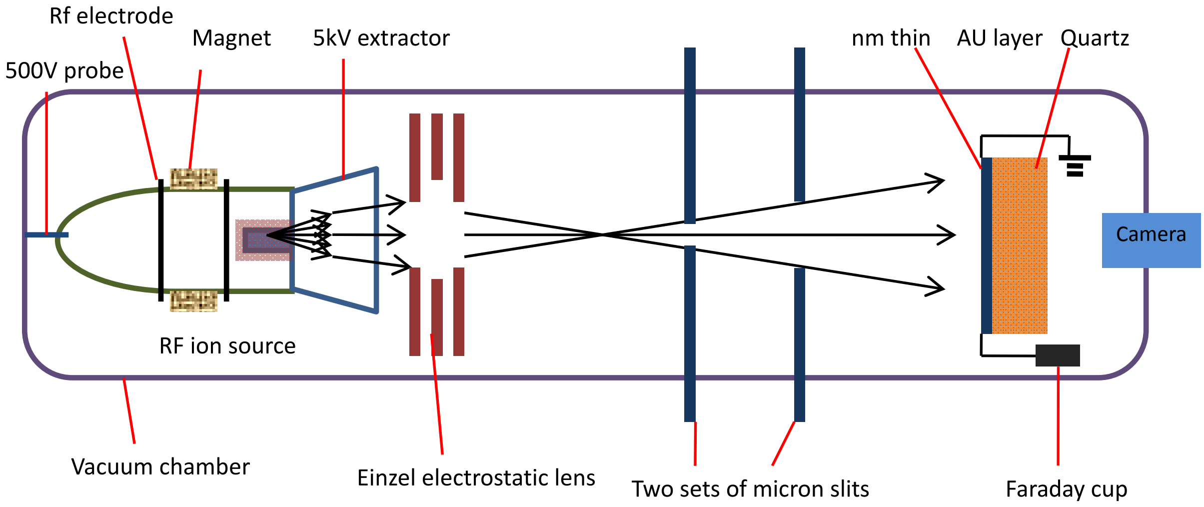


Figure 1. *Simple test bench schematics for RF ion source testing.*

The different ion sources will first be tested at low voltages of 2 – 10 kV. This will give fast and reliable information about the operation of these sources, therefore the high voltage dome (see Fig. 2) will only be designed when both the RF ion source and electron impact ion source work satisfactory at low voltage.

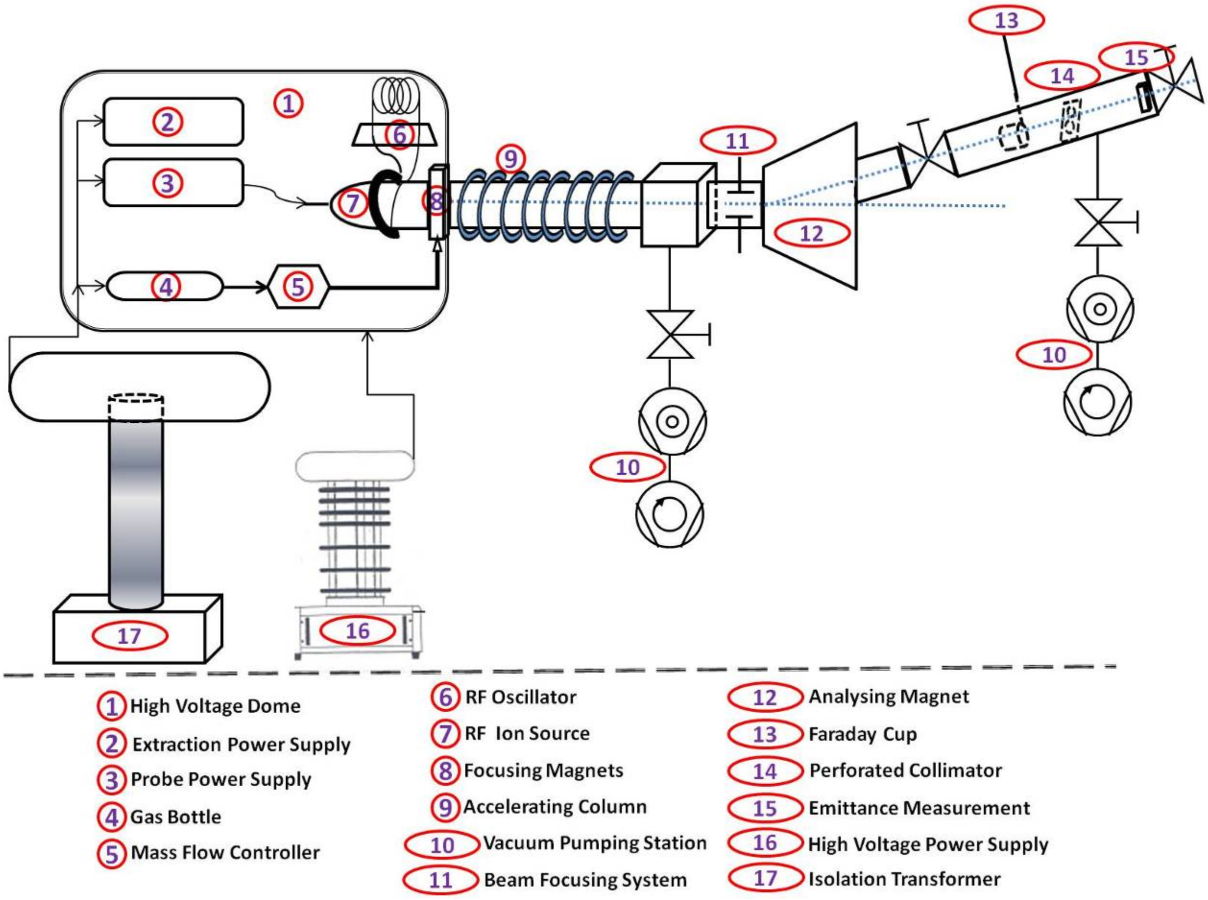


Figure 2: *Schematic diagram of Ion-Source Test Bench along with the low energy ion accelerator set-up.*

Figure 3 shows the actual photograph of the ion-source test bench set up at CIBA, NUS. In accordance to the initial proposal, we have built a test bench for optimizing the existing RF ion source, currently used in the CIBA accelerator. Currently the RF ion source is operational in the test bench at a beam energy up to 3 kV.

The required gas, to be ionized in the RF ion source, is fed through a coarse needle valve regulator (marked as *Gas handling system* in Fig. 3). An oscillating RF voltage, operated at around 100 MHz, is capacitatively coupled onto the gas filled quartz tube (marked as *RF Ion Source* in Fig. 3). The plasma, inside the quartz tube, is generated by the coupling of RF power of a few hundred watts to a gas at a pressure of 10^{-4} to 10^{-5} mbar (as measured in the *vacuum chamber*). The plasma is biased relative to the extraction canal and the ions are extracted through the narrow canal, with a variable extraction voltage of 0 to 3 kV. The whole assembly (ion source, power supplies, RF generator etc.) is housed in an insulated enclosure, which is elevated to the required high voltage, generated by a 10 kV solid-state power supply. The extracted beam is accelerated in a column, made with a series of aluminum electrodes separated by insulating rings (marked as *Acceleration column* in Fig. 3). A constant potential drop is maintained across each electrode, through an appropriate resistor arrangement, so as the accelerated ions will reach ground potential on the last electrode.

As a trial run, we managed to operate the ion source and produce nitrogen ions with energy of 2 keV from this test bench set-up. Initially, the potential of the ion-source assembly was raised to about 2 kV with the help of a 3kV isolation transformer. Further to this process, the amount of gas fed, RF power and probe voltage were remotely adjusted, using a fiber optic communication system, to an optimal valve to form the plasma in the ion-source quartz tube. The pressure of such stable plasma was maintained at around 1.5×10^{-6} mbar (as measured in the *vacuum chamber*). Then the positive ions from the ion source were extracted by negatively biasing the plasma with -1 kV. Currently the accelerating column only served as a drift tube for these extracted 2 keV N^+ ions. The ion current of 2 keV N^+ ions was measured to be around 350 nA on a conductive copper plate, positioned at the center of *vacuum chamber*. Later, upon installing a 250 kV isolation transformer, the whole ion-source assembly will be elevated up to 100 kV; thereby enabling the extracted beam to accelerate through the accelerating column to reach its corresponding high energy (<100keV). With beam Emittance measurement, the high energy ions will be subjected to various beam characterization to quantify the performance of RF ion-source. This set-up will further allow us to optimize different ion-source parameter (RF Ion Source: RF power, Gas pressure, Ion-Source Geometry, Probe Voltage, Extraction Voltage etc.) to obtain the deterministic one order higher beam brightness. In future the possibility of adopting this test-bench to evaluate the performance of any futuristic high brightness ion-sources (e.g. NAIS) will be systematically examined.

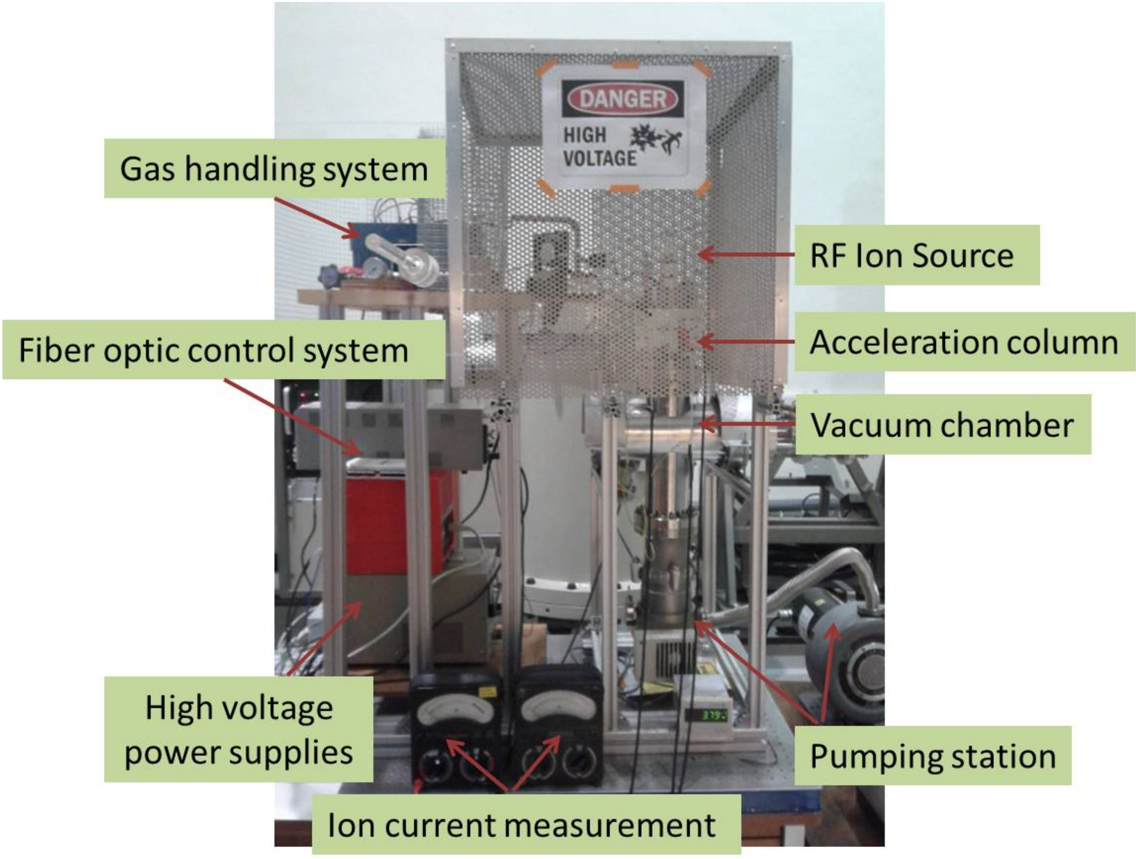


***Figure 3**: Photograph of the existing ion source test setup. The system can presently be pumped down to 10^{-7} mbar. In the left top we see the gas handling system, on the right an RF ion source, at the base there are 2 High Voltage power supplies of 2kV & 5 kV, and on the right bottom the pumping system.*

Fiber optic communication system

During the operation of the ion-source, as stated above, the whole ion-source assembly will be elevated to high potential (~100 kV). With such a large potential difference on this isolated assembly, it becomes lethal for an operator to touch-and-operate the appropriate components. But in practice, to obtain an optimal ion current, the ion-source parameters (RF power, probe voltage, extraction voltage etc.) has to be tuned dynamically. With diligent effort, a solution to this constrain was achieved by controlling the high voltage components remotely using the fiber optic communication system. Figure 4 shows the schematic view of the fiber optic communication system adopted to control the individual components, at the high voltage terminal, remotely in our test-bench setup at CIBA, NUS. The computer codes were developed using Labview Control VI programs to control and diagnose the input and output signals to these components. The combination of two USB extender (local and remote) system (Make: ICRON, Model: USB Ranger® 2224) was used to communicate the required signal from the computer to the data acquisition (DAQ) card (Make: National Instruments, Model: NI-USB-6211) at the high voltage terminal. The communication between the USB extender at the ground terminal and high voltage terminal was achieved through a long Duplex LC connector fiber optic

cable. The signal reaching the remote USB extender, via optic fiber, is then fed to the DAQ card, which controls the individual high voltage power supplies (like RF power, probe voltage, extraction voltage etc.). The present remote USB extender system has the capability to control four different DAQ cards independently, and each DAQ (NI-USB-6211) can control two power supplies simultaneously, thus enabling to control eight power supplies in total at the high voltage terminal. Here in our trial run, we demonstrated the successful operation of probe voltage power supply, at high voltage terminal, using this remote control fiber optic system. With the ion-source assembly elevated to a higher potential of 2 kV, the probe voltage was varied, remotely, between 50 and 500 V using this fiber optic communication system. The increase in probe voltage had a corresponding effect on the extracted ion current (increased from 75 nA to 350 nA). This accomplished methodology used in remote controlling the probe voltage will later be extended to operate other power supplies located at the high voltage terminal, enabling us to obtain an optimal ion current and brightness from the RF ion source.

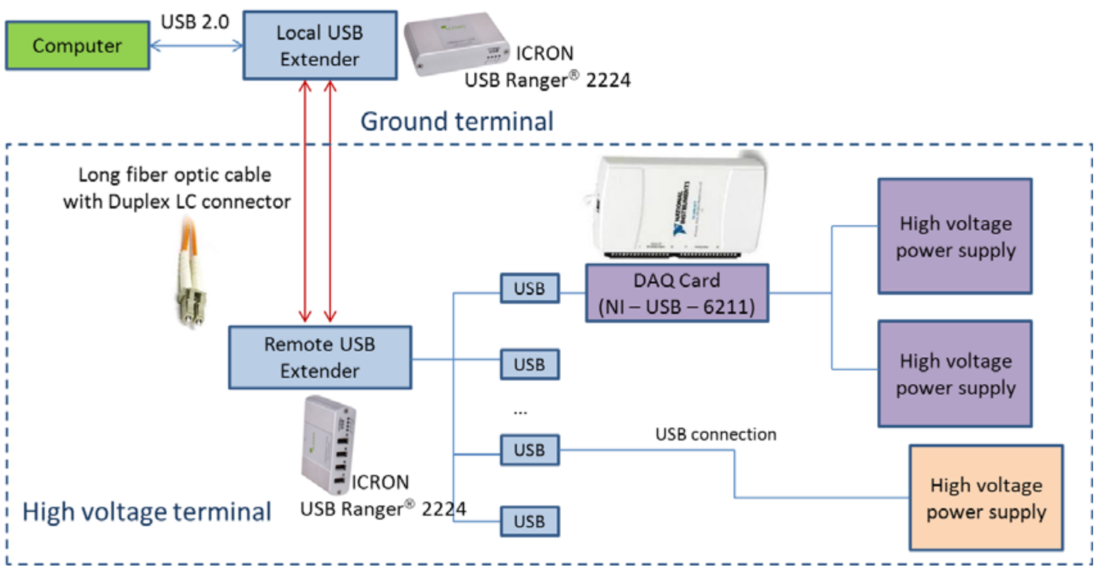


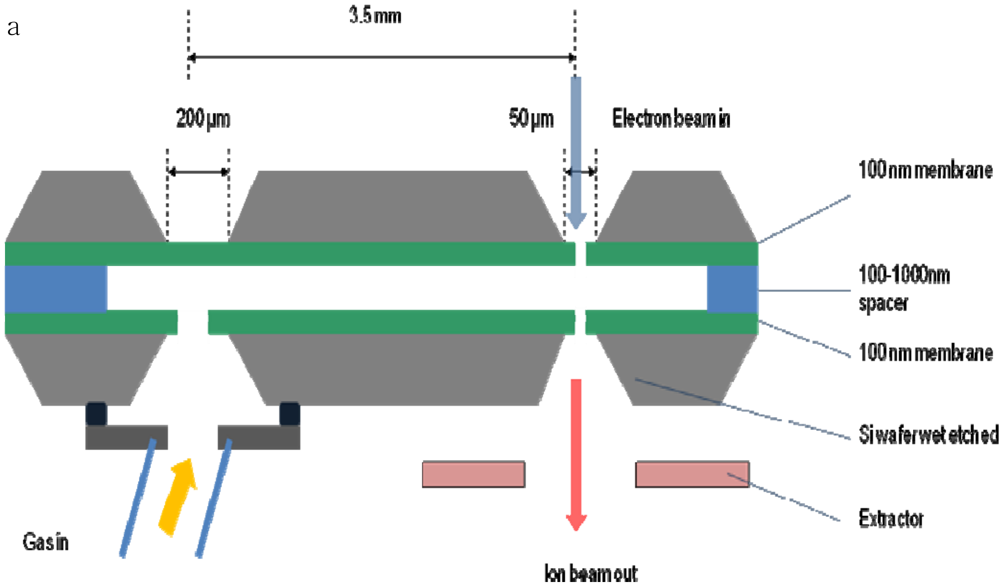
Figure 4: *Schematic of fiber optic communication system used to control the components, at the high voltage terminal, remotely in the ion-source test bench setup at CIBA, NUS.*

The electron impact gas ion source test

A prototype Nano Aperture Ion Source (NAIS) gas chamber has been tested by Prof Kruit, Prof Hagen and a PhD student in Delft for the last year. The experiments have shown an ion beam current of 100-200 pA can be easily achieved. A first calculation shows the ion beam brightness is about $3\text{x}10^4$ A/(m^2 sr V), which is in consistence with the theoretical calculation and comparable to a conventional Gallium Liquid Metal Ion Source (LMIS) for Focused Ion Beam (FIB). To obtain a higher brightness ion beam, a new NAIS gas chamber has been developed in Delft. We have tested in collaboration with Delft a new chip design.

The electron impact gas ion source that is being developed to improve the brightness for MeV proton beam writing applications will be an alternative for the radio frequency (RF) ion sources that are commonly used to provide proton and helium ion beams for lithography and nuclear microscopy applications in MeV accelerators.

The electron impact gas ion source designed in Delft is expected to give much higher reduced brightness, about 10^7 A/m^{2}SrV. Prototypes of the new ion source have been fabricated and tested using a Schottky electron source as injector. The idea is to introduce an electron beam into a small chamber (100-1000 nm), where gas particles are ionized (Figure 5). The positive ions are then extracted by the electric field from an extractor. The ion current is measured by a Faraday cup and pico-amp-meter.



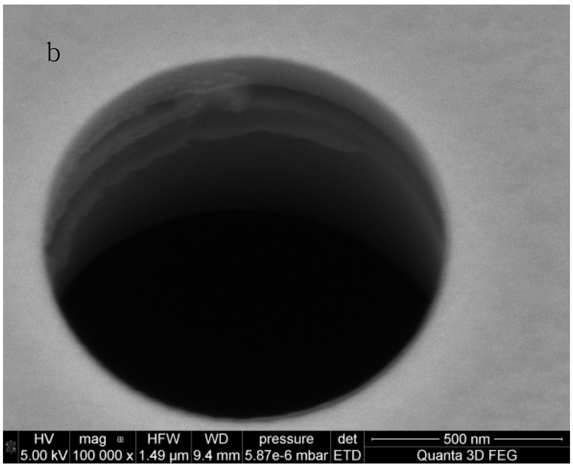


Figure 5: *Electron impact gas ion source. a) A narrow channel path for gas is created by depositing a thin membranes on Si. Two entrances of 50 µm and 200 µm for beam transport and gas inlet are created respectively. b) High magnification of 500 nm apertures surrounding the gas ionization chamber.*

Figure 6 shows the test setup with the NAIS chip mounted in the sample holder (Delft test setup). The NASI gas chamber is sitting in a SEM chamber, where an electron beam is introduced into the gas chamber to bombard the gas particles for ionization. The induced ion beam current and brightness depend on a few parameters that can be varied for test, which includes the gas feed-in pressure, the bias voltage between the two membranes in the gas chamber, the electron energy and electron beam current. Currently the new NAIS gas chamber needs further improvements and is still under inspection.

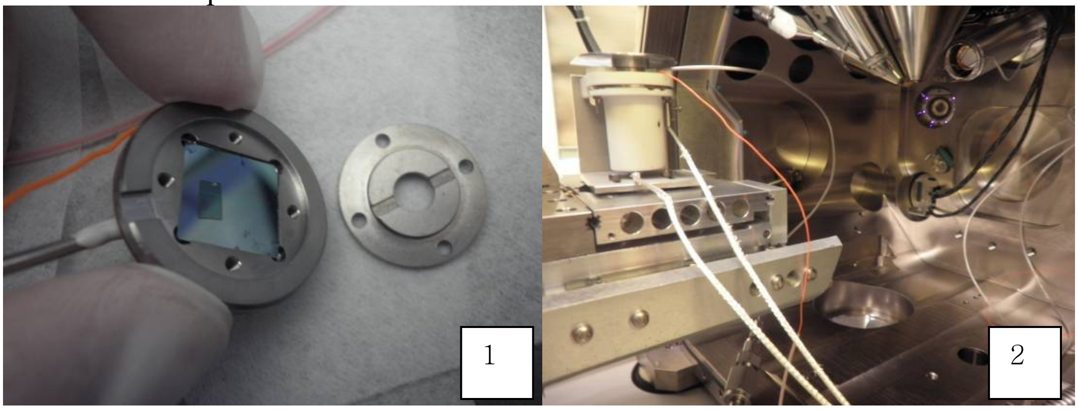


Figure 6. *1) NAIS gas chamber in sample holder; 2) gas chamber mounted in sample holder and sits on SEM stage in chamber.*

With a 1 keV electron beam, the extracted ion currents for helium, air, argon and Xenon gases have been obtained and are shown in figure 7 as functions of gas inlet pressure [5].

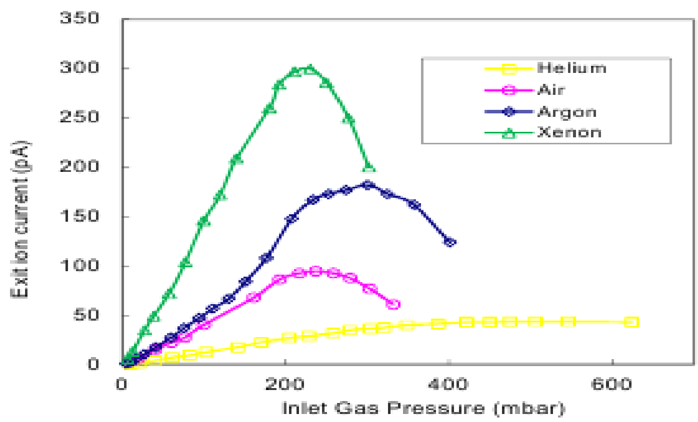


Figure 7: *The ion beam current outputs from the NAIS source with different gases. At an electron current of 14 nA the ion current varies as a function of pressure and ion type. The current drops with higher gas pressure due to the reduced mean free path.*

The electron impact gas ion source design calculations

The ion beam current is a function of the electron current, ionization path length and electron ionization cross-section. The ion beam reduced brightness therefore depends on the **gas ionization chamber geometry**, ion type, electron beam energy and **brightness**. Our current focus is on the theoretical evaluation of

conventional electron emitters as injector for this electron impact gas ion source and the gas ionization chamber geometry. We are comparing tungsten hair-pin, LaB6 and Schottky electron sources with hydrogen gas for 10 nm to 10 μm aperture sizes and 100 nm to 1 μm ionization path length (ie membrane spacing). Increasing the path length will yield higher ion current densities at the expense of ion energy spread. The electron beam is optimized for maximum current density in the chamber by considering the electron source brightness and system optical aberrations, without including coulomb interactions [5]. Figure 8 shows the calculated ion beam reduced brightness as a function of aperture size.

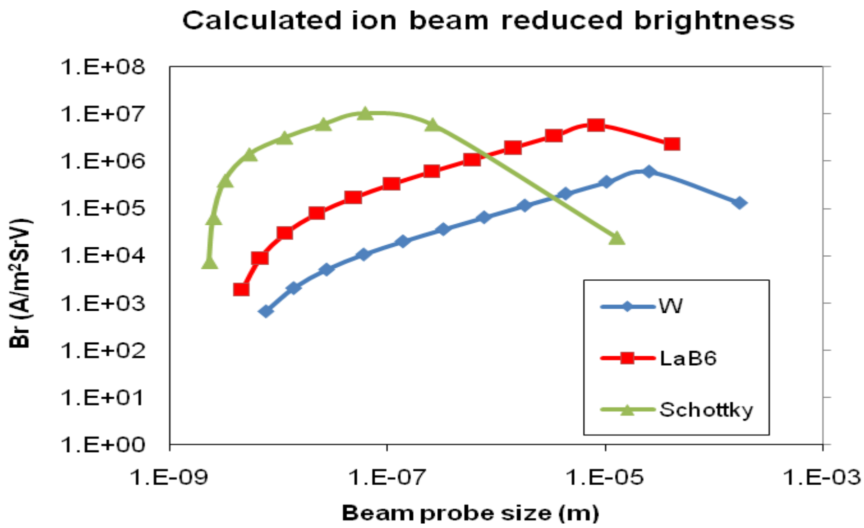


Figure 8: *Ion reduced brightness of hydrogen gas with tungsten hair-pin, LaB6 and Schottky electron sources as a function of aperture size. The ionization path length is 100 nm and the electron beam energy is 1 keV.*

The proposal of a compact high brightness PBW system is based on the current 2nd generation PBW beam line at CIBA, NUS [4]. To achieve writing speeds comparable to commercial EBL systems, we aim for a proton beam current of 1 pA with a beam reduced brightness of 0.2-5× 10^6 A/m^{2}SrV. Protons from the NAIS ion source will be accelerated up to 200 keV and focused in the target chamber using either a spaced Russian quadruplet lens configuration or a Spaced triplet lens configuration.

The design of the ionization chamber for the three electron injectors of the electron impact gas ion source includes a few parameters e.g. path length, gas pressure and ionization chamber dimensions. The ionization path length is proposed to be as 1 μm with an inlet gas pressure of 120 mbar for all the three electron injectors. The output proton beam current is maximized avoiding ion-gas molecule collisions. The gas leakage through this ionization chamber is on the order of 10^{-10} - 10^{-9} mbar l/s. Comparing the three electron injectors, the Schottky electron column shows the greatest potential for a high brightness (2.5× 10^6 A/m^{2}SrV) and high beam current (0.1-0.2 pA) for fast PBW. The effects of Coulomb interactions on the beam spot size are estimated for this system. The trajectory displacement due to Coulomb interactions is about 2 nm at the target plane, indicating the possibility of achieving sub-10 nm proton beam. We used Particle Beam Optics Laboratory 3.0 (PBO Lab)

simulation to find an optimal quadrupole lens configuration and beam spot size. Simulation result shows that the beam from an aperture of 60 nm can be demagnified down to 7.5 (2) × 7.5 (2) nm^2 by the Russian quadruplet configuration. Similarly a 200 nm beam can be focused down to 1.5 (1.5) × 5.5 (4) nm^2 using the spaced triplet configuration in X and Y direction. The outcome of the calculations and simulations of compact PBW systems using the three electron injectors are summarized in Table 1.

Table 1: Summary of the ionization chamber designs and specifications for a 200 keV compact PBW system using Schottky , LaB_6 and Tungsten hair-pin electron injectors.

Lithographic probe	**Schottky-injector PBW**			**LaB6-injector PBW**		**W-injector PBW**	
Gas inlet pressure (mbar)	120			120		120	
Ionization path length (µm)	1			1		1	
Gas leakage rate (mbar l/s)	2.5×10^{-10}			1.0×10^{-9}		1.0×10^{-9}	
Chamber Aperture Diameter (nm)	60	60	200	60	200	60	200
Lens configuration	Russian Quadruplet	Spaced Triplet	Spaced Triplet	Russian Quadruplet	Spaced Triplet	Russian Quadruplet	Spaced Triplet
Proton beam size (nm2)*	7.5×7.5	1×2	1.5×5.5	7.5×7.5	1.5×5.5	7.5×7.5	1.5×5.5
Maximum proton source emission current (pA)	600	600	3000	12	1000	0.6	60
Maximum proton beam current (pA)	0.2	0.2	0.1	0.005	0.01	0.0003	0.004
Proton beam reduced brightness (A/m^2SrV)	2×10^6	2×10^6	1×10^6	6×10^4	1×10^5	3×10^3	5×10^3
Writing time (second)	5	5	10	200	100	3333	250

*the proton beam size in this table does not include the Coulomb interactions.

Next test stage of the electron impact ionization process

A miniature field emission electron microscope is being evaluated for testing the electron impact gas ionization test chips in NUS Singapore. In this microscope the acceleration voltage can be varied from 0 – 5 kV. We are planning to adjust the specimen chamber of this electron microscope (see Figure 9 & 10) to house the NAIS gas chip and Faraday cup. This will allow us to determine the most suitable electron beam energy as well as possible alternative configurations.

Figure 9 *Miniature Field Emission Electron microscope which is under study to be used to test electron impact ionization gas chambers in NUS.*

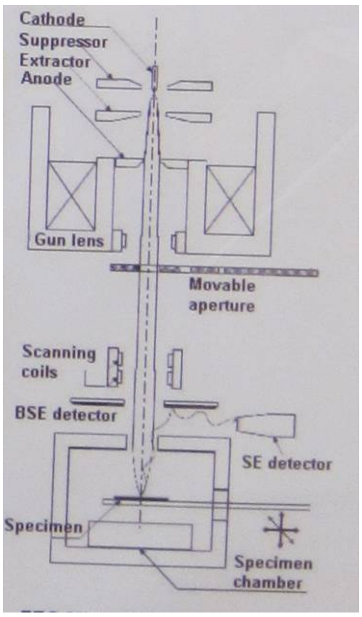


Figure 10 *Schematics of the miniature field emission electron microscope.*

List of Publications:

Since the system is still under development we have been studying different designs. At the 3 Beams conference the work on the design of the new electron impact ion source was selected as an invited poster. We are currently preparing a paper on this work which we plan to submit to JVSTB. The PI has also published several papers based on earlier grants from the US air force: **AOARD 07-4017, AOARD 06-4004 & AOARD 05-4037**

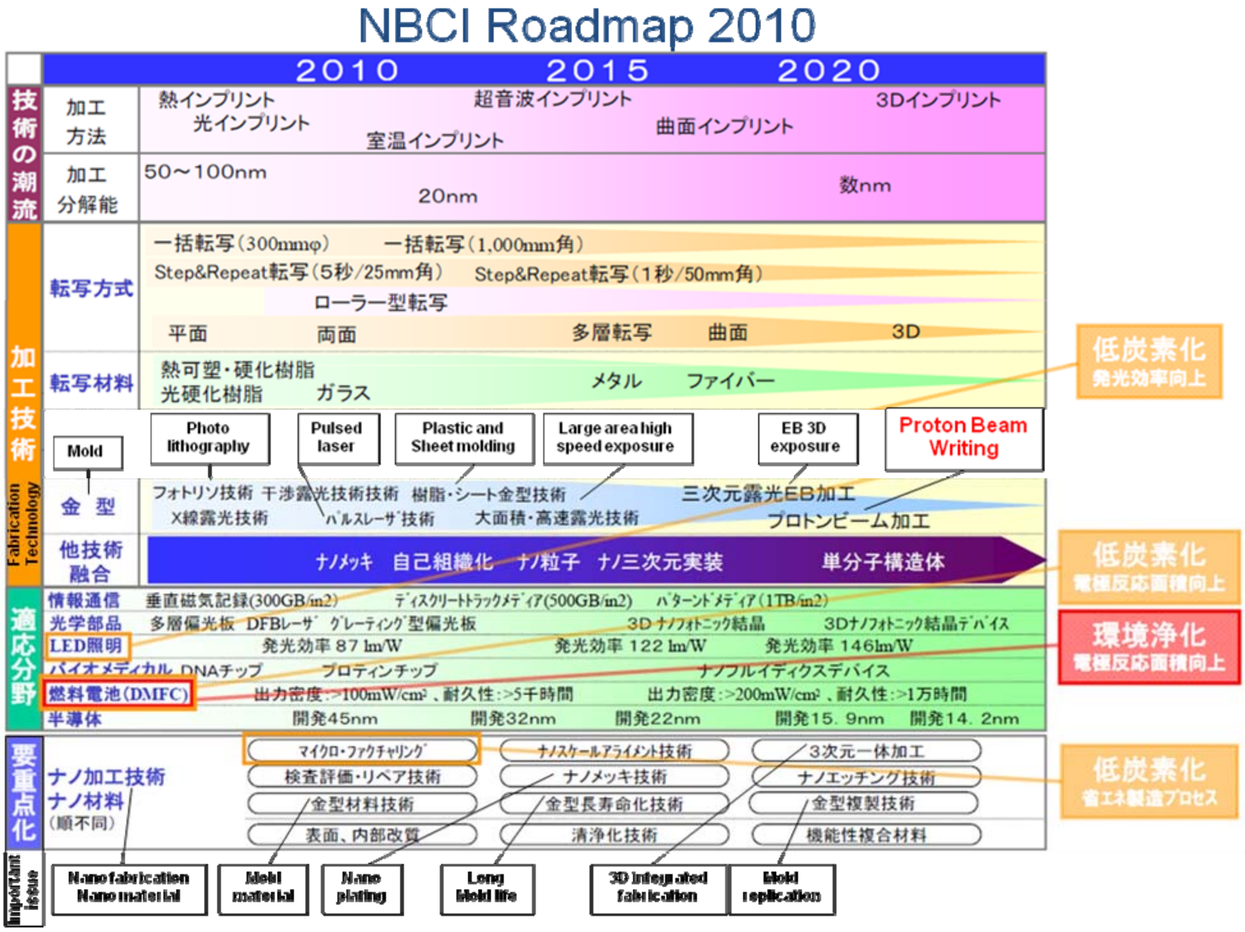
Journal Publications:

1) Injector evaluation in an electron impact ion source for high brightness proton beam writing, Nannan. Liu [1,a)], David S. Jun[2)], Cornelis W. Hagen, Pieter Kruit[2)] P. Santhana Raman[1)] and Jeroen A. van Kan, In preparation
2) Preliminary lithographic structures attained from the next generation proton beam writing facility, Yao Yong, P. Santhana Raman, J.A. van Kan, Submitted JVSTB
3) Improved beam spot measurements in the 2^{nd} generation proton beam writing system, Yao Yong[a)], Martin W. van Mourik[b)], P. Santhana Raman[a)] and Jeroen A. van Kan[a], Submitted to Nucl. Inst. Meth B
4) High throughput fabrication of disposable nanofluidic lab-on-chip devices for single molecule studies, JA van Kan, C. Zhang, P. Malar and J.R.C. van der Maarel, Biomicrofluidics **6** (2012) 036502-1, 036502-9
5) The second generation Singapore high resolution proton beam writing facility, J.A. van Kan, P. Malar, and Armin Baysic de Vera, Review of Scientific Instruments **83** (2012) 02B902-1 02B902-3.
6) The Singapore high resolution single cell imaging facility, Frank Watt, Xiao Chen, Armin Baysic De Vera, Chammika C N Udalagama, Ren Minqin, Jeroen A van Kan, Andrew A Bettiol, Nuclear Instruments & Methods in Physics Research Section **B** *in press*.
7) Proton beam writing nanoprobe facility design and first test results, J.A. van Kan, P. Malar, Armin Baysic de Vera, Xiao Chen, A.A. Bettiol and F. Watt, Nuclear Instruments & Methods in Physics Research Section **A 645** (2011) 113-115.
8) Proton Beam Writing a platform technology for high quality three-dimensional metal mold fabrication for nanofluidic applications, J.A. van Kan, P.G. Shao, Y.H. Wang and P. Malar, Microsystem Technologies **17** (2011) 1519-1527.
9) Exposure parameters in proton beam writing for KMPR and EPO Core negative tone photoresists, M.D. Ynsa, P. Shao, S.R. Kulkarni, N.N. Liu, J.A. van Kan, Nuclear Instruments & Methods in Physics Research Section **B 269** (2011) 2409–2412

<u>Conference Organization/presentations:</u>

1. **Session Chair of two sessions** at the 22nd International Conference on the Application of Accelerators in Research and Industry (5 – 10 August 2012, Fort Worth, Texas, USA, CAARI has over 500 participants).
2. **Two Invited review presentation: 2012,** 22nd International Conference on the Application of Accelerators in Research and Industry (5 – 10 August 2012, Fort Worth, Texas, USA), Title: 1) *Precise mold fabrication using proton beam writing for Cell and DNA manipulation. 2) Next Generation MeV Proton Beam Focusing; What is required for sub 10 nm 3D lithography?*
3. **Invited Oral presentation**: **2012** International Conference on Nuclear Microprobe Technology and applications (Lisbon, Portugal). Title: *The 2nd Generation Proton Beam Writing Facility; Results and Outlook.*
4. Invited poster presentation at the the 56th International Conference on Electron, Ion and Photon Beam Technology & Nanofabrication, 28 May – 1 June 2011, Hawaii, USA. Title: *Electron impact gas ion source development; evaluation of different electron injection sources*, N. Liu, J.A. van Kan, D. Jun, C.W. Hagen, P. Kruit.
5. Oral presentation: at the the 56th International Conference on Electron, Ion and Photon Beam Technology & Nanofabrication, 28 May – 1 June 2011, Hawaii, USA. Title *First Lithography results obtained with the 2nd generation MeV proton beam writing facility*.
6. 2011 **Chair of section BB** of the International Conference on Materials for Advanced Technologies ICMAT 26/06/11–1/07/11, Singapore (ICMAT 2700 participants).
7. **Invited Oral presentation**: **2011** February at the Ion-beam Induced Nanopatterning of Materials, Bhubaneswar, India. Title *Nickel injection mould fabrication via proton beam writing and UV lithography for fluidic chip applications*
8. **Invited Oral presentation**: **2010** July at the 8th Charged Particle Optics conference, Singapore. Title Proton *beam nano-probe technology and applications*
9. Oral presentation: June 2011 at the International Conference on Materials for Advanced Technologies 26/06/11 – 1/07/11, Singapore. Title *Next Generation Proton Beam Nano-probe Technology for Proton Beam Writing.*
10. Poster presentation at 36th MNE conference, September 2010, Genoa, Italy On "*Mold fabrication using PBW for nano fluidic and DNA analysis*"
11. Poster presentation at ICNMTA, July 2010, Leipzig Germany. On "*The Singapore next generation proton beam writing facility"***.** This poster got an honourable mentioning at the conference.

Appendix 1



Publications: See attached zip file.

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2 *The National University of Singapore high energy ion nano-probe facility: Performance tests,* F. Watt, J.A. van Kan, I. Rajta 1, A.A. Bettiol, T.F. Choo, M.B.H. Breese, T. Osipowicz, Nuclear Instruments and Methods in Physics Research **B 210** (2003) 14–20.

3 Proton Beam Writing of 3D Nanostructures in Hydrogen SilsesQuioxane J.A. van Kan, A.A. Bettiol and F. Watt, *Nano letteres* **6**, (2006), 579.

4 *The second generation Singapore high resolution proton beam writing facility*, J.A. van Kan, P. Malar, and Armin Baysic de Vera, Review of Scientific Instruments **83** (2012) 02B902-1 02B902-3.

5 P. Kruit, M. Bezuijen and J.E. Barth, J. Appl. Phys., **99**, 024315 (2006).